

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Serial No: 10/613,002  
Filed: July 2, 2003  
Title: DISPERSE AZO DYE MIXTURES  
.....

Art Unit: 1714  
Examiner: Patrick D. Niland

Hon. Commissioner of Patents & Trademarks  
Washington, D. C. 20 231

DECLARATION (Rule 132)

Sir:

I, Dr. Clemens Grund from Hattersheim, declare: I am a chemist and a citizen of the Federal Republic of Germany, residing at Diedenbergerstr. 7, 65795 Hattersheim, Federal Republic of Germany.

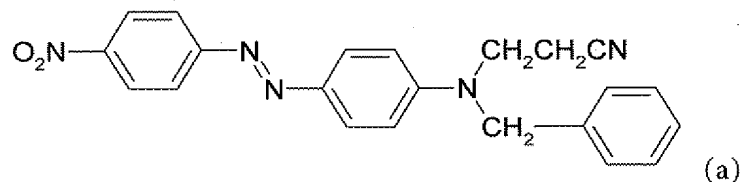
Since completing my studies at the University Freiburg im Breisgau in Germany and having taken my doctor's degree in 1990 at the same University, I have been employed as a research chemist in the field of organic chemistry by BASF Aktiengesellschaft, Ludwigshafen, Germany. In October 2000 BASF transferred its activities in the textile dyestuff field to DyStar and since then I am employed by DyStar Textilfarben GmbH & Co. Deutschland KG in Frankfurt, Germany. From 1997 until 2003 I was head of a production site in Omuta, Japan and since 2003 and until today I am "Head of Product Innovation Synthetics & Wool". I have had adequate professional experience in the field to which patent application Serial No. 10/613,002, filed July 2, 2003, pertains and which was filed by Manfred Hoppe, Kiyoshi Himeno and Ryouichi Sekioka.

I further declare:

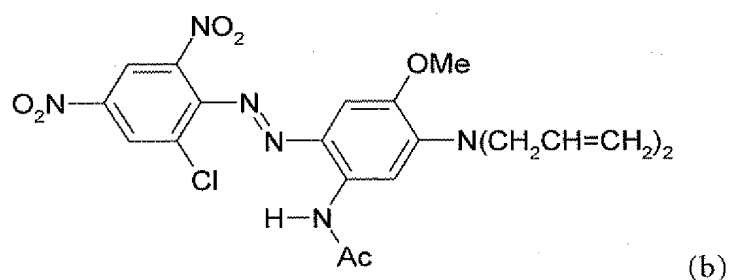
In order to demonstrate that the dye mixtures according to the present application are not obvious over the teachings of the prior art the tests described below have been carried out under my personal guidance and supervision.

**TEST A****I) Dye Mixtures**

A) Dye mixture A containing 5 parts of the dye of formula a,

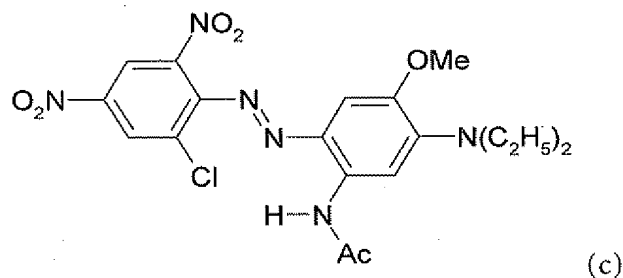


and 39 parts of the dye of formula b,



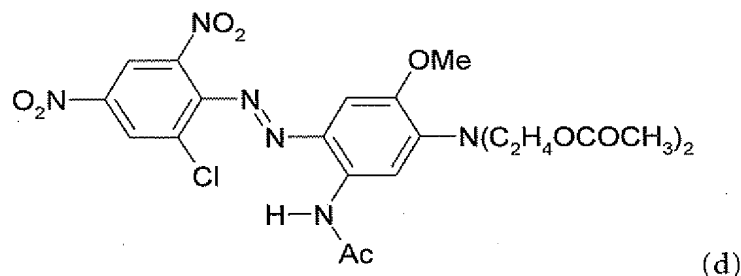
according to U.S. Patent Application Serial No. 10/613,002.

B) Dye mixture B containing 5 parts of the dye of formula a (see above) and 39 parts of the dye of formula c



according to U.S. Patent Application Serial No. 10/613,002.

C) Dye mixture C containing 5 parts of the dye of formula a (see above) and 39 parts of the dye of formula d



according to prior art.

## II) Determination of the dye mixture's sensitivity to change of pH

The sensitivity of dyeings obtained with dye mixtures A, B and C to change of pH value was determined. For that purpose dyeings at two different depth of shade standards and at different pH values were produced and compared.

To obtain the depth of shade standards the following amounts of dye mixtures (in each case calculated as 100%) were used in form of 2% dye dispersions:

Dye mixture	depth of shade standard 1 (bright shade)	depth of shade standard 2 (dark shade)
A	0.3060	1.0174
B	0.6066	2.1380
C	0.3350	1.1000

Dyebaths at pH values of 4.5, 5.5, 6.5 and 7.5 were produced for each dye mixture and for each depth of shade standard by using the amounts of dye mixture given above, 1 g/l of a dispersing agent (Setamol BL) and sodium dihydrogen phosphate and disodium hydrogen phosphate, respectively, as buffer. The pH values were adjusted using acetic acid and sodium carbonate, respectively.

The dyebaths thus obtained were used to dye in each case 5 g of a polyester fabric at 130°C for 60 minutes. The dyeings were after treated as usual and dried and then evaluated colorimetrically by means of reflection color measurement.. The dyeings obtained at pH 5.5 were used as standard and set to 100%. The following results were obtained:

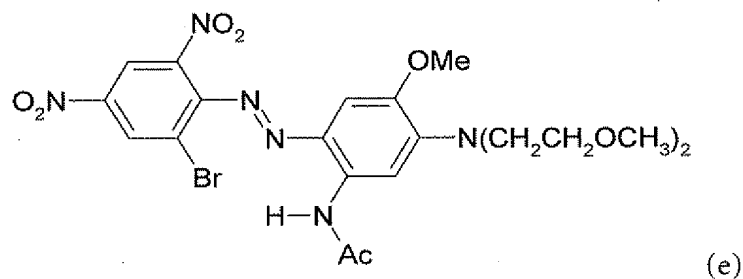
Dye mixture	pH 4.5	pH 5.5	pH 6.5	pH 7.5
	depth of shade standard 1			
A	99.5	100	97	94.4
B	99.4	100	99.2	98.3
C	100	100	93	55

	depth of shade standard 2			
A	100	100	100	100
B	99.1	100	95.6	95
C	99.8	100	94.3	77.4

## **TEST B**

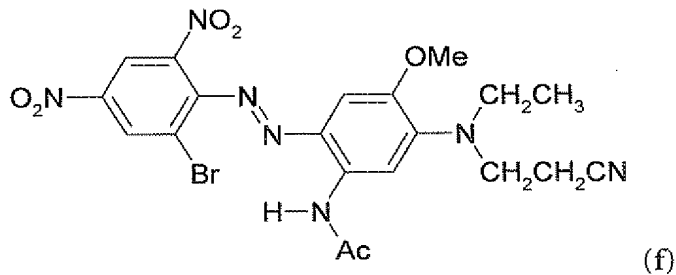
### I) Dye Mixtures

A) Dye mixture D containing 5 parts of the dye of formula a (see TEST A above) and 39 parts of the dye of formula e,



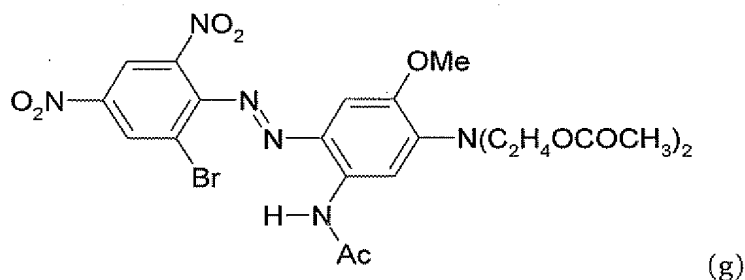
according to U.S. Patent Application Serial No. 10/613,002.

B) Dye mixture E containing 5 parts of the dye of formula a (see TEST A above) and 39 parts of the dye of formula f



according to U.S. Patent Application Serial No. 10/613,002.

C) Dye mixture F containing 5 parts of the dye of formula a (see TEST A above) and 39 parts of the dye of formula g



according to prior art.

## II) Determination of the dye mixture's sensitivity to change of pH

The sensitivity of dyeings obtained with dye mixtures D, E and F to change of pH value was determined. For that purpose dyeings at a certain depth of shade standard and at different pH values were produced and compared.

For that purpose, dyebaths for each dye mixture were produced at pH values of 4.5, 5.5, 6.5 and 7.5. The pH values were adjusted using acetic acid and sodium carbonate, respectively.

The dyebaths thus obtained were used to dye in each case 5 g of a polyester fabric at 130°C for 60 minutes. The dyeings were after treated as usual and dried and then evaluated colorimetrically by means of reflection color measurement.. The dyeings obtained at pH 5.5 were used as standard and set to 100%. The following results were obtained:

Dye mixture	pH 4.5	pH 5.5	pH 6.5	pH 7.5
D	100	100	98	94
E	100	100	99	91.9
F	100	100	98,6	78,6

## RESULTS OF TESTS A AND B

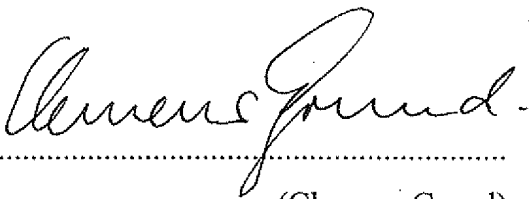
The tests show that the dyeings obtained with the inventive dye mixtures A and B and D and E, respectively, do not differ or differ only little from the standard. With other words they are

not at all or only little sensitive to change of pH value. In contrast to this, the deviation obtained with dye mixtures C and F, respectively, according to prior art at pH 7.5 is considerable. These dyeings are thus sensitive to change of pH value to a very big extent.

I further declare that I understand the contents of this Declaration, that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at Frankfurt

This 11th day of August 2006

  
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(Clemens Grund)